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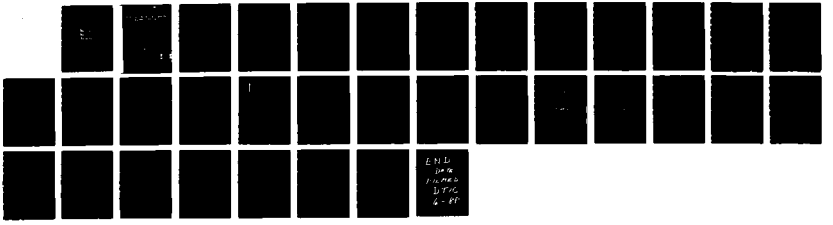

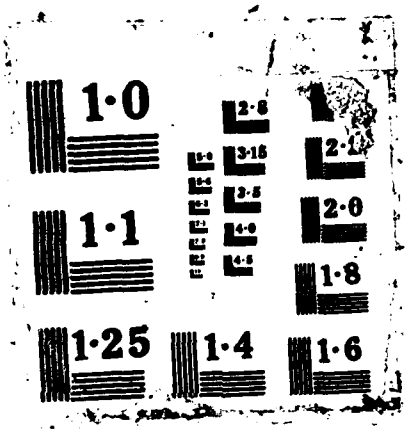
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EVALUATION OF COAL-DERIVED EDS MID-DISTILLATE

INTERIM REPORT
BFLRF No. 227

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A cooperative demonstration program between industry and the Department of Energy yielded a sizable quantity of coal-derived, mid-distillate fuel producted by the Exxon Donor Solvent (EDS) process. Eighty-three 55-gallon drums of this product were made available to the Belvoir Fuels and Lubricants Research Facility at Southwest Research Institute for evaluation through the U.S. Army Belvoir Research, Development and Engineering Center, Ft. Belvoir, VA. The EDS mid-distillate was characterized in the laboratory for chemical and physical properties, and evaluated in a medium-speed diesel engine. Due to the low cetane number, toxicity, poor storage/thermal stability, and incompatibility with elastomers, this fuel was not evaluated as received in the normal operation of diesel engines. A 69/31 blend of the EDS mid-distillate and a petroleum-derived diesel fuel was used with relative success in a medium-speed diesel engine. A similar blend evaluated in single-cylinder diesel engine burned less efficiently than the petroleum fuel.

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FOREWORD

This work was performed at the Belvoir Fuels and Lubricants Research Facility, Southwest Research Institute, under DOD Contract No. DAAK70-85-C-0007, Work Directives No. 8 and 27. The project was administered by the Fuels and Lubricants Division, U.S. Army Belvoir Research, Development and Engineering Center, Ft. Belvoir, VA 22060-5606, with Mr. F.W. Schaekel, STRBE-VF, serving as Contracting Officer's Representative. This report covers the period of performance from October 1982 through December 1984.

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Mr. J.A. Russell provided and interpreted the thermal oxidation test data, and Dr. George Lee interpreted the gas chromatographic analyses data. The authors wish to acknowledge the assistance given by Mr. J.W. Pryor and the Belvoir Fuels and Lubricants Research Facility editorial group in the preparation of this report.

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I. INTRODUCTION

In a cooperative program between industry and the Department of Energy, Exxon Research and Engineering Company demonstrated, over a 2-year period, the technology for the Exxon Donor Solvent (EDS) process in a 250-ton-per-day coal liquefaction pilot plant. At the completion of this program, DOE made available drum quantities of EDS mid-distillate for testing and evaluation. In concert with one of the goals of the U.S. Army Belvoir Research, Development and Engineering Center to evaluate alternative fuels such as liquefied coal, 83 55-gallon drums of EDS mid-distillate were received at Belvoir Fuels and Lubricants Research Facility (BFLRF) at Southwest Research Institute for evaluation.

Certain conditions were agreed upon prior to shipment of this material to Belvoir Fuels and Lubricants Research Facility. These conditions are listed in the EDS Process Samples Agreement, which is included in the appendix.

II. OBJECTIVE

The objective of this program was to perform physical and chemical characterizations necessary to determine the suitability of EDS mid-distillate as a diesel fuel and to evaluate it as fuel for medium-speed diesel engines.

III. APPROACH

The sample was characterized by laboratory tests. Blends of the EDS mid-distillate and a reference DF-2 petroleum fuel were used to generate power in the medium-speed, eight-cylinder Enterprise stationary diesel engine located at BFLRF, and evaluated in a single-cylinder, two-stroke diesel engine.

IV. RESULTS AND DISCUSSION

A. Properties

The results of the EDS mid-distillate characterization are shown in TABLE 1. Similar data for the reference DF-2 (Cat 1-H) are shown for comparison. The low API gravity

TABLE 1. Properties of EDS Middle Distillate and Reference Diesel Fuel

Test Method	Test Values	
	EDS, AL-12168-SP-F	Cat 1-H Typical
Gravity, °API, D 1298	14.8	34.8
Specific gravity, 16/16°C, D 1298	0.9672	0.8509
Distillation, °C, D 86 (°C, D 2887)		
IBP	212 (166)	213
10% recovered	230 (216)	243
20% recovered	237 (233)	253
50% recovered	269 (284)	274
90% recovered	377 (397)	322
EP	> 406* (>511)	349
Recovered, vol%	95	99
Residue, vol%	4	1
Cetane number, D 613	21	50
Cloud point, °C, D 2500	Too dark	-9
Pour point, °C, D 97	-17	-9
Accelerated stability, mg/100 mL, D 2274	77.9	0.47
Ash, wt%, D 482	0.001	0
Carbon residue, wt%, D 524	1.21 (on total sample)	0.10 (on 10% bottoms)
Elemental analysis, wt%		
Carbon, D 3178 modified	89.00	86.32
Hydrogen, D 3178 modified	9.94	12.86
Nitrogen, Chemiluminescence	0.17	ND**
Sulfur, D 2622	0.01	0.41
Oxygen (by difference)	0.88	None
Phenols, wt%, UOP Method 262-59	1.01	ND
Thiophenols, wt%, UOP Method 262-59	<0.01	ND
Carbon type, wt%, UV Spectroscopy		
Mono-aromatics	24.2	6.0
Di-aromatics	12.3	7.4
Tri-aromatics	1.7	1.1

* The end point distillation temperature was beyond the range of the thermometer used for this test.

** ND = Not determined

(high specific gravity) indicates a high concentration of aromatics. The high concentration is confirmed by the percentages of mono-, di-, and tri-aromatic hydrocarbons. The distillations performed show the presence of high boiling material, which would be considered above the boiling range of petroleum-derived middle distillates, such as No. 2 diesel fuel and No. 2 burner fuel.

The low cetane number (21) precludes the material from being used neat as a diesel fuel in medium- or high-speed compression-ignition engines. The high insolubles obtained in the accelerated stability test indicate poor storage stability with respect to insoluble sediment formation.

B. Response to Additives

Cetane improver, octyl nitrate, was added at the levels of 0.5 and 1.0 vol%, and the cetane numbers measured by ASTM D 613 as summarized in TABLE 2.

TABLE 2. Cetane Number Response of EDS Mid-Distillate to Cetane Improver

Fuel Sample	Cetane Number
EDS mid-distillate neat	21.0
EDS mid-distillate plus 0.5 vol% octyl nitrate	24.8
EDS mid-distillate plus 1.0 vol% octyl nitrate	28.1

This response did not compare favorably with that of a petroleum diesel fuel, which increased seven numbers with the addition of only 0.1 vol% octyl nitrate, or with a shale-derived middle distillate, which increased five numbers with the addition of 0.1 vol% of the same cetane number improver additive.

C. Thermal Stability Evaluation

The EDS mid-distillate was evaluated by the Jet Fuel Thermal Oxidation Test (JFTOT), ASTM D 3241 procedure, at 260°C. No pressure drop across the filter was observed during the test period; however, the JFTOT tube visual deposit rating was >4, TDR spun deposit rating was 21 at tube position 28, and the TDR spot deposit rating was 27 at tube position 28. These data are those shown for Test No. 2 in TABLE 3. Based on this test, the EDS mid-distillate has poor thermal stability characteristics.

TABLE 3. Thermal Oxidation Fouling Tester Data

Test No.	Fuel Code	Description	Elastomer	Sump Temp, °C*	Aluminum Tube Temp, °C	ΔP mm Hg/min	Tube Visual Rating	Max TDR Spun/Location	Max TDR Spot/Location	Elastomer Swell, %	Elastomer ΔH Shore A
0	AL-12168	EDS**	Buna 70	66	260	124/146	Peacock	23/48	26/48	+31.8	-3.8
1	AL-12168	EDS	None	66	260	4/150	>4	21/36	29/36	--	--
2	AL-12168	EDS	None	24	260	0/150	>4	21/28	27/28	--	--
3	AL-12168	EDS	Viton	66	260	125/107	>4	26/41	27/41	+0.12	+2.5
4	AL-12168(45%) AL-11804(55%)	EDS Cat 1-H***	Buna 70	66	260	125/18	>4	>50/--	>50/--	+11.66	+2.6
4A	AL-12168(45%) AL-11804(55%)	EDS Cat 1-H	Buna 70	66	260	125/32	>4	>50/--	>50/--	+11.43	+0.70
5A	AL-12168(20%) AL-11804(80%)	EDS Cat 1-H	Buna 70	66	260	125/12	>4	>50/--	>50/--	+7.41	-3.4
5B	AL-12168(20%) AL-11804(80%)	EDS Cat 1-H	Buna 70	66	260	125/17	>4	>50/--	>50/--	+6.44	+2.2
6	AL-11804	Cat 1-H	Buna 70	66	260	125/51	3	23/44	25/44	+2.45	0.5
7	AL-11804	Cat 1-H	None	66	260	125/67	4-Peacock	21/39	23/39	--	--

* Sump temperature maintained during 3-hours pretest time and 150-min standard D 3241 test flow of 3 mL/min to heater test tube.
** EDS = Exxon donor solvent middle distillate.
*** Cat 1-H = Reference No. 2 diesel fuel.
-- = Not Applicable.

D. Thermal Oxidation Fouling Test

The EDS mid-distillate and blends with a reference petroleum diesel fuel, Cat 1-H, were evaluated in the Thermal Oxidation Fouling Tester (TOFT). This apparatus is essentially the same as that used in ASTM D 3241 (JFTOT) procedure with additional capabilities of variable fuel reservoir configurations, reservoir heating, variable fuel flow rate, and a variety of heater tube configurations.(1)* For this evaluation, the standard JFTOT configuration was used but the fuel was heated and stirred with a magnetic stirrer in the fuel reservoir to maintain a 66°C temperature. The membrane filter was removed from the prefilter assembly. The Cat 1-H reference fuel is considered to be a high-deposit fuel for lubricant testing in the 1G2 and 1H2 single-cylinder engine tests.

Test coupons of elastomers were placed in the heated sump to evaluate the effect of the fuels and blends on the elastomers. The data obtained for both EDS mid-distillate, Cat 1-H, and blends are shown in TABLE 3. The presence of Buna N-70 in the sump at 66°C during tests with EDS mid-distillate accelerated the pressure developed across the test filter element, which was the standard 17 μm, nominal screen opening, stainless-steel mesh filter. At 24°C sump temperature, no pressure drop was observed in 150 minutes with the neat fuel. Viton® in the hot sump caused accelerated pressure differential increase, but the Viton® specimen swelled only a fraction of one percent. All the tests produced high visual and TDR ratings. The swell percentage of the Buna N-70 specimens shows a correlation with the concentration of EDS middle distillate in Cat 1-H, which is plotted in Fig. 1. The change in hardness of the elastomer specimens did not follow any pattern. It is apparent that the EDS middle distillate has a significant effect on the swell property of Buna N-70, but not on Viton®.

The time elapsed for filter plugging to occur, as indicated by a pressure differential (DP) of 125 mm Hg, during the tests has been plotted in Fig. 2 against the concentration of EDS mid-distillate in Cat 1-H. The EDS mid-distillate test went 146 minutes before plugging occurred, and the Cat 1-H test took 51 minutes. All the blends took 32 minutes or less to plug the filters, indicating some incompatibility between the EDS mid-distillate and the Cat 1-H diesel fuel.

* Underscored numbers in parentheses refer to the list of references at the end of this report.

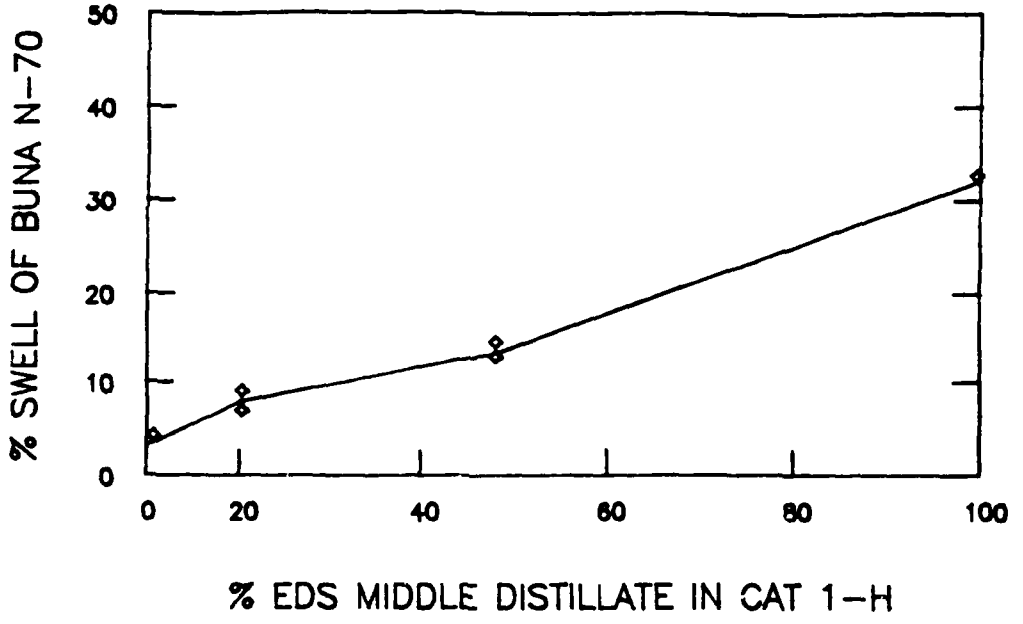


Figure 1. Effect of EDS on Buna N-70 swell

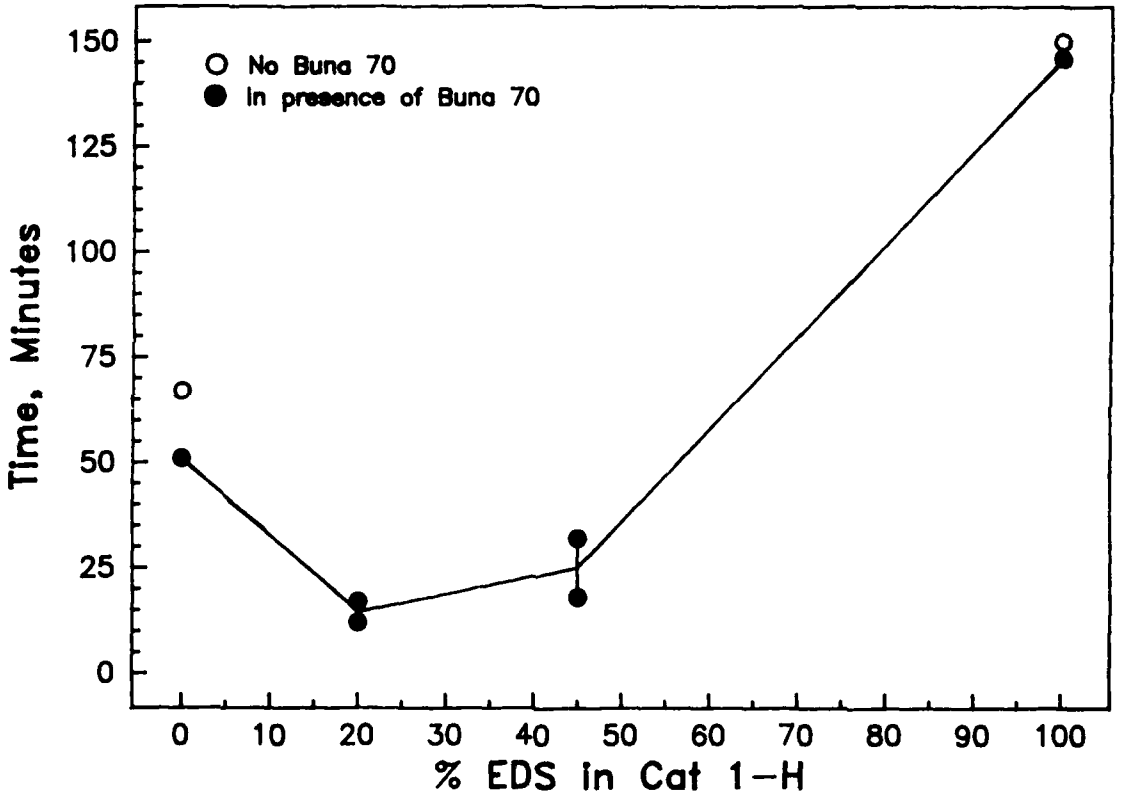
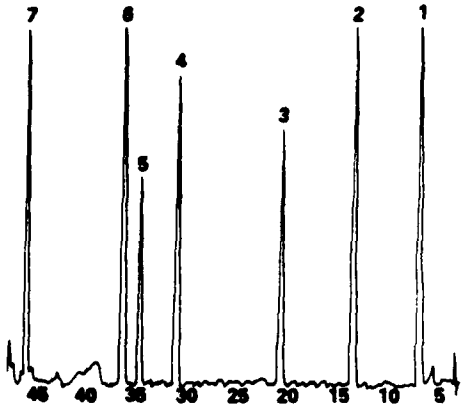


Figure 2. ASTM D 3241 test filter plugging time ((DP = 125 mm Hg) as a Function of EDS Concentration in Cat 1-H, Sump at 60°C)

E. Analysis of Individual Phenolic Components in Coal Liquids

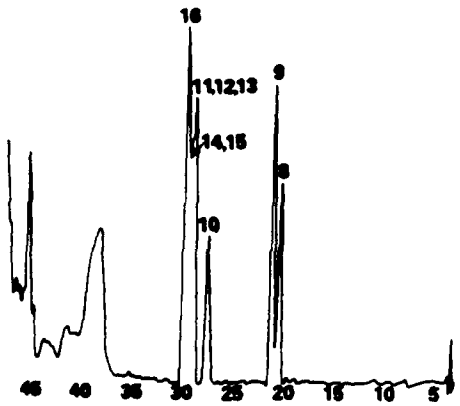
In an effort to characterize individual phenolic compounds in the EDS mid-distillate, a procedure published by Chao and Suatoni was emulated.(2) This procedure called for initial separation of the polar species from the main portion of the liquid with column chromatography employing a short packed column. This separation was followed by acetonitrile desorption of the trapped compounds and analysis by high-performance liquid chromatography (HPLC). The results of this analysis are shown in Fig. 3 for the EDS extract along with sixteen known phenol standards. Figs. 3a and 3b show the results of the separate GC analyses of the listed compounds. The unlabeled peaks are due to impurities in the individual phenol standards. Fig. 3c shows the result of the HPLC analysis of the acetonitrile extract of the EDS. There appears to be a large amount of unresolved material in the extract, which does not allow baseline resolution as in Figs. 3a and 3b. The lack of resolution and difficulty of interpretation led to an attempt at direct analysis by GC(FID) (Fig. 4). Although the resolution and interpretability improved somewhat, they were still deemed insufficient for definitive analysis.

Certain other methods for analysis of phenols, e.g., EPA Method 604 (3), and methods for acylation derivatization (4) call for use of derivatization agents, usually containing fluorine, and GC analysis with an electron capture detector (ECD). Both EPA Method 604 and the GC-ECD method were attempted after extraction of the phenols with aqueous NaOH. This procedure calls for shaking the EDS mid-distillate sample with an 1N aqueous NaOH solution. After separation, the aqueous layer is acidified, and the solution is shaken with a suitable solvent, e.g., benzene, to remove the freed phenols. The organic layer is separated, dried, derivatized (3-4), and analyzed by GC-ECD. In both methods, the signals were enhanced due to the use of the ECD. All the sixteen standard compounds appeared to be individually resolved for the first time in this series of experiments. The retention times of the standard materials were determined for each compound separately, by injecting 1 µL of a solution of 0.5 µL benzene and 0.5 µL of each compound into a column described as follows: (EPA Method 604) (3)



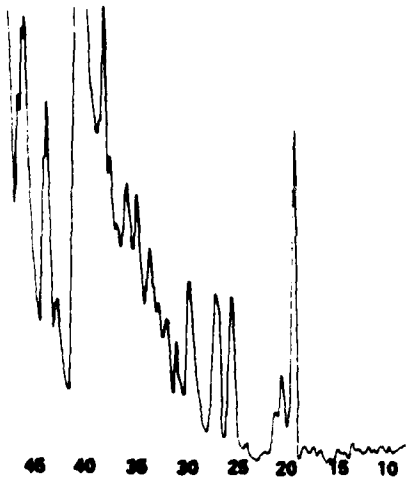
Peak No.	Description	Retention Time (min)
1	resorcinol	7.4
2	phenol	13.4
3	o-cresol	20.6
4	2-ethylphenol	31.0
5	2, 3, 5-trimethylphenol	34.7
6	2-isopropylphenol	36.3
7	pentachlorophenol	45.9

a. Composite 7 Phenols Standards



Peak No.	Description	Retention Time (min)
8	p-cresol	20.5
9	o-cresol	21.1
10	3, 4-dimethylphenol	27.5
11, 12, 13	3, 5-dimethylphenol 2, 3-dimethylphenol 2, 6-dimethylphenol	28.9
14, 15	2, 5-dimethylphenol 2,4-dimethylphenol	29.22
16	4-ethylphenol	29.6

b. Composite 9 Phenols Standards

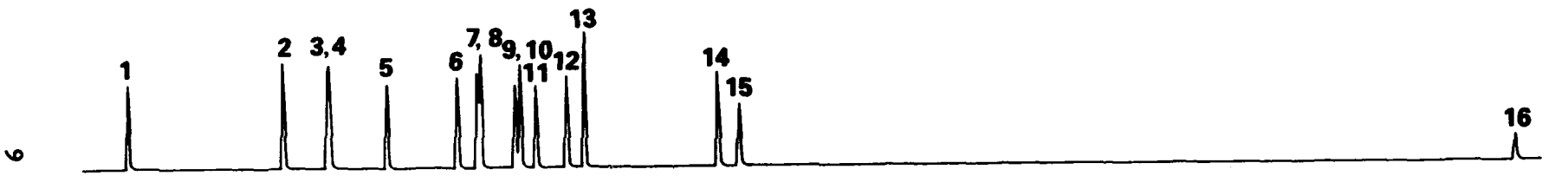


c. Acetonitrile Extract of EDS

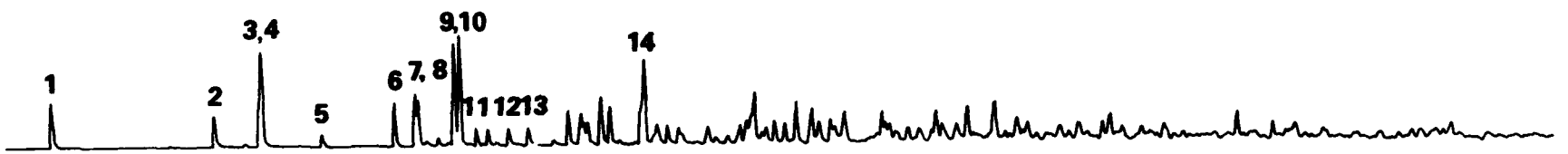
Figure 3. HPLC analysis of phenolic materials
(abscissa measured in minutes, ordinate is relative refractive index detector response)

CHROMATOGRAPHIC ANALYSIS OF 16 PHENOL STANDARDS

No.	Standard	Retention Time	No.	Standard	Retention Time
1	phenol	20.49	10	p-ethylphenol	28.17
2	o-cresol	23.54	11	3, 5-dimethylphenol	28.47
3, 4	m/p-cresol	24.42	12	3, 4-dimethylphenol	29.07
5	2, 6-dimethylphenol	25.56	13	2-isopropylphenol	29.39
6	o-ethylphenol	26.91	14	2, 3, 5-trimethylphenol	32.01
7	2, 4-dimethylphenol	27.29	15	2-isopropylphenol	32.44
8	2, 5-dimethylphenol	27.39	16	pentachlorophenol	47.55
9	2, 3-dimethylphenol	28.05			



a. Standard Materials



b. EDS Acetonitrile Extract

Figure 4. Gas chromatographic analys of phenolic materials

Column:	Pesticide Column - 1/4 in. x 6 ft.
Flow:	60 mL/min
Carrier Gas:	5% CH_4/Ar bal.
Detector:	250°C
Injector Port:	150°C
Column Temperature:	Initial temperature, 50°C
	Initial hold, 2 min
	Program rate, 4°C/min
	Final temperature, 100°C

TABLE 4 lists the retention times determined for the sixteen standard compounds. The gas chromatogram for the EDS mid-distillate shown in Fig. 5, was generated by injecting 1.2 µL of 100 µL EDS caustic extract, taken to dryness, and mixed with 0.5 µL benzene. A comparison of the peaks in the gas chromatogram with the retention times shown in TABLE 4 permitted an estimation of the presence or absence of the phenolic compounds in the mid-distillate extract, as indicated in the TABLE 4 column headed "Analysis Results."

TABLE 4. Phenolic Species in EDS
(From Fig. 4)

Peak No.	Standard	Retention Time, minutes*	Analysis Results
1	Phenol	2.8	Probably present
2	0-cresol	4.7	Probably present
3	m-cresol	5.2	Probably present
4	p-cresol	5.4	Probably present
5	2,6 xylenol	5.7	Probably absent
6	0-ethylphenol	6.3	Probably present
7	2,5 xylenol	7.1	Probably present
8	2 isopropylphenol	7.2	Probably present
9	2,4 xylenol	7.3	Probably present
10	p-ethylphenol	7.4	Probably present
11	3,5 xylenol	7.6	Probably present
12	2,3 xylenol	8.3	Probably absent
13	3,4 xylenol	8.7	Probably present
14	Resorcinol	9.7	Probably present
15	2,3,5 Trimethylphenol	10.4	Probably present
16	Pentachlorophenol**	22.4	Uncertain**

* Retention times determined from chromatograms for standard compounds run under same conditions as the unknown sample.

** The presence of pentachlorophenol, indicated as possible on the chromatogram, was questioned by Exxon.

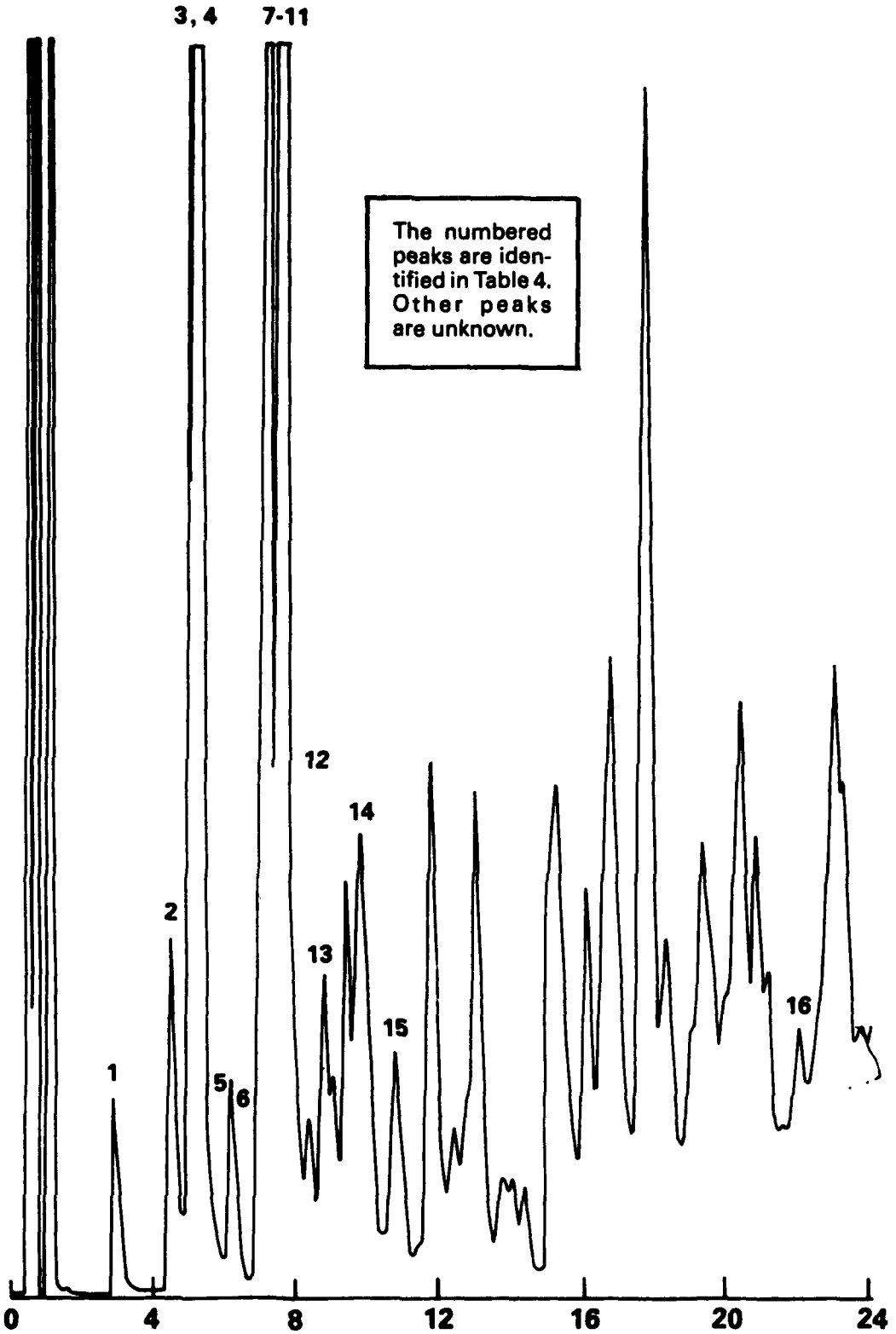


Figure 5. Gas chromatogram for EDS extract

F. Injector Fouling Bench Test

A 55/45 vol% blend of reference diesel fuel and EDS mid-distillate was evaluated in the injector fouling bench test (IFBT).(5) The IFBT apparatus was operated on the blend for 41 hours without any injector nozzle fouling. The final deposition ratings--8.40 and 4.70 tip and shaft demerits, respectively--were only slightly higher than the Cat 1-H reference fuel tip (8.00) and shaft (4.20) demerits. However, the deposit buildup on the injector tip using the EDS blend appeared to be heavier and softer than the deposits from the reference fuel. Additionally, a thin film of deposits that had not been seen before was noted on the shaft of the injector pintle; however, this deposit did not impair the pintle motion as the pop-off pressure and spray remained good throughout the test.

G. Evaluation of EDS in the Enterprise DSM-38 Diesel Engine

The fuel requirements for the Enterprise DSM-38 diesel engine, as described in the instruction manual for this engine, are shown in TABLE 5. The properties of the EDS fuel and of a blend of the EDS material with a reference diesel fuel (Cat 1-H) are also provided in TABLE 5. The EDS material fails to meet many of the requirements for the Enterprise engine including API gravity, Conradson carbon residue, distillation, and cetane number. The blend of 55 percent Cat 1-H and 45 percent EDS was prepared based on a mathematically estimated cetane number of 35 assuming linear addition. The determined value, 36, as shown in the table, and other properties are much closer to the Enterprise engine fuel requirements.

Mixtures of EDS mid-distillate and a reference diesel fuel were evaluated in the Enterprise diesel engine. This engine drives an electrical generator which, in turn, produces power that is fed into the local electrical grid. The engine is adjusted to produce 420 to 470 kW during operation.

The engine was operated first on the diesel fuel, then on a trial blend of the EDS-containing mixture. Due to the higher density of the EDS component, the engine fuel control system had to be readjusted to reduce the fueling rate in order to maintain the desired power output rate. Other than this adjustment, no changes were made to the engine.

TABLE 5. Requirements and Fuels for Enterprise DSM-38 Diesel Engine

	Fuel Requirements*	AL-12168-SP-F EDS	55% Cat 1-H 45% EDS
Viscosity, cSt at 37.8°C	20.4 max	--	--
Viscosity, cSt at 40°C	--(1)	--	3.56
Gravity, °API	28 to 38	14.8	26.5
Sulfur, wt%	2.0 max	0.01	--
Copper corrosion, 3 hr at 100°C	Pass	--	1b
Conradson carbon residue, wt%	0.20 max	1.5	0.39
Ash, wt%	0.10 max	0.001	--
Water and sediment, vol%	0.50 max	--	0.2
Flash point, °C	65 min	--	--
Pour point, °C	**	-17	--
Distillation, °C			
10% point	266 max	230	238
90% point	371 max	377	340
End point	385 max	406 at 94%	406 at 96%
Cetane number	35-45	21	36

* Fuel requirements for Enterprise DSM-38 Engine taken from the Instruction Manual.
** 5.5°C below coldest fuel oil temperature.
(1) No requirements or not determined.

After the trial run with the EDS material had been completed successfully, an extended run of about 24 hours was conducted with the EDS-containing blend. The results of the three brief trials and the extended operation are summarized in TABLE 6.

Based on these limited results, no difficulties or short-term penalties appear to be associated with use of this type of fuel blend in medium-speed diesel engines. There was essentially no difference in fuel performance, and no operational difficulties were observed. If anything, the operator commented that the engine seemed to perform better with less visible smoke with the test mixture than with the reference diesel fuel.

TABLE 6. Operation of Enterprise Engine on Various Test Fuels

Fuel	Duration of Operation, hr	Fuel Consumed, lb	Total Output, kW-hr	Avg Power Output Rate, kW-hr/lb ($X 10^{-3}$)
• Cat 1-H	1.5	362	1.1	3.04
• 60% Cat/ 40% EDS	1.3	310	0.9	2.90
• Cat 1-H	1.5	348	1.0	2.87
• 69% Cat/ 31% EDS*	22.3	4920	16.0	3.25

* Blend ratio used was lower than desired due to a blending error.

Two trial runs with the Cat 1-H reference fuel gave average power output values of 3.04 and 2.87 kW-hr/lb ($X 10^{-3}$), which is reasonable repeatability.

H. Evaluation in a Single-Cylinder Diesel Engine

In addition to the Enterprises tests, the 55-percent Cat 1-H/45-percent EDS blend, AL-12284-F, was evaluated in a single-cylinder, two-stroke diesel engine. The engine was a Detroit Diesel 3-53 two-cycle, direct-injection engine modified to operate on one cylinder and instrumented for cylinder pressure measurement phased with crankangle. The engine was operated at two speeds, 1000 and 1200 rpm. At each speed, the EDS blend was evaluated at a Cat 1-H comparable power level. At each test point, 100 cycles of cylinder pressure data were acquired at 1-degree intervals, then ensemble averaged to obtain a representative cycle. From the pressure histories, a heat release analysis was performed to calculate combustion parameters for comparison with the base fuel Cat 1-H.

The performance and combustion parameters for each test point are shown in TABLE 7. The indicated specific fuel consumption (ISFC) values indicate an 11-percent increase at 1000 rpm, and a 17-percent increase at 1200 rpm for the EDS blend. The apparent combustion efficiencies, which are a measure of the engine's ability to convert chemical energy into heat, reveal lower efficiencies for the EDS blend at both speeds. The

TABLE 7. Performance and Combustion Parameters of Test Fuels at 1000 rpm and 1200 rpm

	1000 rpm		1200 rpm	
	Cat 1-H	EDS Blend*	Cat 1-H	EDS Blend*
Speed, rpm	1001	1001	1202	1201
Load, kg-m	7.19	7.27	6.64	6.22
Fuel Flow, kg/hr	2.54	2.77	2.77	3.04
Indicated Power, kW	9.92	9.74	11.77	11.02
ISFC, kg/kW-hr	0.256	0.284	0.235	0.275
Heat Input, J/inj	1808.8	1912.9	1640.8	1751.1
Peak Pressure, MPa	9.448	9.470	8.982	8.620
Peak Pressure Rise, kPa/deg	1022.0	1541.0	789.4	1389.0
Peak Heat Release Rate, J/deg	125.1	192.8	91.5	175.9
Cumulative Heat Release, J	1198.9	1189.9	1188.8	1146.5
Apparent Combustion Efficiency, %	66.3	62.2	72.5	65.5
Indicated Thermal Efficiency, %	32.9	30.5	35.8	31.5
Ignition Delay, deg	6.2	8.1	8.0	9.0
Centroid Phasing, deg	188.5	188.8	192.2	192.0
Centroid Magnitude, J/deg	21.43	37.38	16.67	27.43
Sensitivity, deg	20.3	18.7	22.1	21.0

* Blend consisted of 55-percent Cat 1-H/45-percent EDS.

indicated thermal efficiencies, a measure of the engine's ability to convert chemical energy to mechanical energy, are also lower at both speeds for the EDS blend.

An examination of the heat release rate diagrams reveals qualitative differences in how fuel burns in the engine. Fig. 6 is a comparison of the heat release rates at 1000 rpm for the EDS blend and Cat 1-H fuel. The longer ignition delay is evident in the diagrams, which can be attributed to the lower cetane number of the blend. As a result of the

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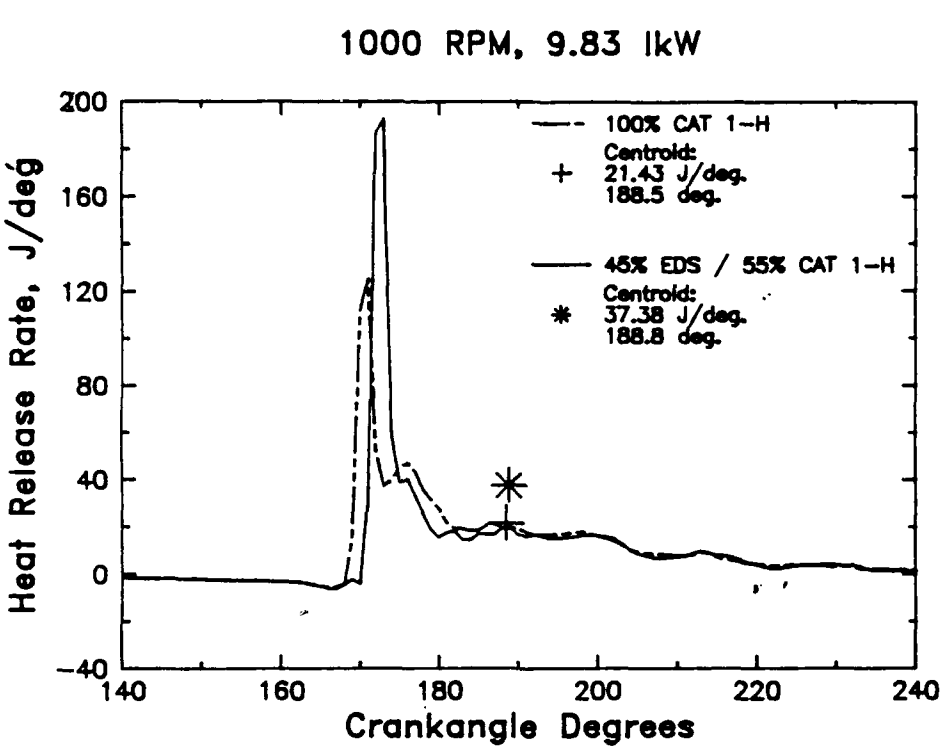

Figure 6. Heat release rate comparison of Cat 1-H and 55-percent Cat 1-H/45-percent EDS at 1000 rpm

longer ignition delay, more fuel is in the premixed vapor phase prior to ignition. After ignition occurs, the premixed fuel burns at an instantaneous, rapid rate, causing a high rate of pressure rise and audible knock. This premixed mode of combustion is the dominant mode with the EDS blend, which reveals lower levels of diffusion-controlled combustion rates when compared to the Cat 1-H burning diagram. The centroid parameters are a method of quantifying how fuel burns in the engine. They are the coordinates of the center of area bounded by the heat release rate diagram. The larger centroid magnitude for the EDS blend indicates an increase in the premixed mode of combustion, due to a lower cetane number. The larger centroid phasing can be attributed to the longer ignition delay. The sensitivity is the relationship of the centroid phasing to ignition, and tends to decrease with larger amounts of premixed mode combustion.

Fig. 7 is a comparison of the heat release rates at 1200 rpm for the EDS blend and Cat 1-H fuel. The longer ignition delay of the EDS blend is revealed, along with the

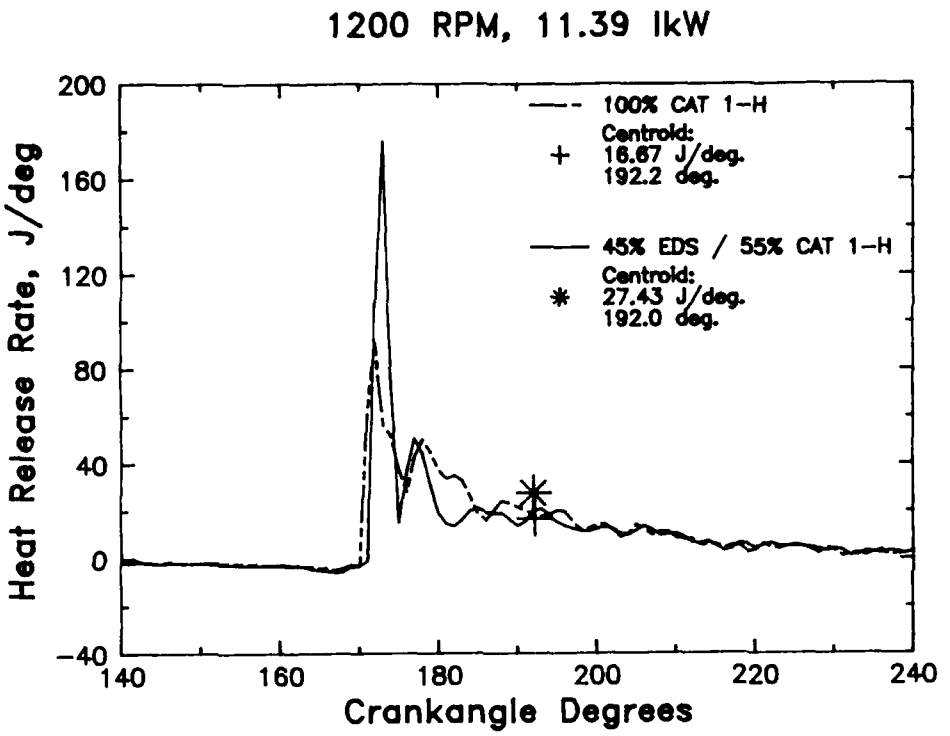

Figure 7. Heat release rate comparison of Cat 1-H and 55-percent Cat 1-H/45-percent EDS at 1200 rpm

associated larger premixed burning spike and the reduced levels of diffusion-controlled burning. Although the diffusion mode of combustion for the EDS blend contributes less to the overall burning than it does for Cat 1-H, it appears to be more significant than it was at 1000 rpm. The larger centroid magnitude reflects the increased premixed mode of combustion, although it is proportionally lower than it was at 1000 rpm when compared to the Cat 1-H values. This lower magnitude can be attributed to the higher diffusion-controlled combustion rates with the EDS blend at 1200 rpm. The centroid phasing is shorter for the EDS blend, which indicates the large premixed burning rate had a greater effect on the center of area than the ignition delay. The correlation of the sensitivity with increased premixed combustion is evidenced by the decrease in sensitivity for the EDS blend at 1200 rpm.

In summation, the 55-percent Cat 1-H/45-percent EDS blend burns less efficiently in the two-stroke direct injection engine than Cat 1-H. This lower efficiency is evidenced by increased fuel consumption, decreased combustion efficiency, and decreased thermal efficiency. The EDS blend also exhibits a much "harsher" combustion, revealing much

higher peak pressure rise and peak heat release rates. This condition leads to heavy audible knock, which is detrimental to the durability of the engine. The heat release rate analysis indicates the EDS blend burns primarily in the premixed combustion mode, instead of the more efficient diffusion-controlled combustion mode.

I. Sediment From EDS Middle Distillate

While using the EDS mid-distillate, AL-12168-SP-F, it was observed that it contained a fine solid material in suspension. This material was separated from the EDS by dilution with 50 percent n-heptane and centrifuging. The separated sediment was examined by scanning electron microscope and appeared to be mainly carbonaceous in nature.

Elemental analysis by combustion for carbon and hydrogen (modified ASTM D 3178), by chemiluminescence for nitrogen, and by X-ray fluorescence spectrometry for sulfur, iron, silicon, chlorine, calcium, chromium, and manganese gave the results shown in TABLE 8.

TABLE 8. Analysis of Solids in EDS Middle Distillate

Element	Wt%
Carbon	80.80
Hydrogen	5.89
Sulfur	0.69
Nitrogen	3.51
Iron	0.14
Silicon	0.07
Chlorine	1.12
Calcium	0.36
Chromium	0.02
Manganese	0.03
Oxygen - by difference	7.37

V. CONCLUSIONS

The use of EDS mid-distillate as received in diesel engines would not be practical for several reasons uncovered by this evaluation, including:

- Low-ignition quality as measured by cetane number, which was not improved significantly by use of additives.
- Toxic nature due to the presence of polynuclear aromatics, phenolic and nitrogen compounds.
- Poor storage and thermal stability, which may lead to filter plugging and fuel system deposits if used in an engine.
- Incompatibility with elastomers commonly found in engine systems.

When the EDS mid-distillate was blended with over 50 percent of a petroleum diesel fuel, and evaluated in the Injector Fouling Bench Test and in the Enterprise medium-speed diesel engine, problems anticipated with the use of neat EDS were minimized. The engine seemed to run smoothly and operate well with the fuel mixture.

The EDS blend with petroleum fuel burned less efficiently than the petroleum fuel alone and exhibited a "harsher" combustion.

VI. DISPOSITION OF UNUSED SAMPLE

The balance of the EDS mid-distillate not used in this evaluation, 76 full and sealed drums and one partial drum, were returned to the Exxon Research and Engineering Company as agreed in the initiation of this program.

VII. ADDITIONAL INVESTIGATIONS

Hydrogenation of the EDS mid-distillate was performed in a pilot plant, located at Southwest Research Institute, to produce a series of three coal-derived test fuels for combustion studies. The feedstock was obtained through the Department of Energy, and the three products differed in properties depending on the severity of processing. The resulting products were stable, had low heteroatom content, and had considerably improved cetane numbers; however, the latter were lower than the cetane numbers for most petroleum-derived middle distillates.(6)

VIII. LIST OF REFERENCES

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APPENDIX
EDS PROCESS SAMPLES AGREEMENT

October 25, 1982

EDS Process Samples Agreement

CDR
U.S. Army Mobility Equipment Research
and Development Command
ATTN: DRDME-GL
Fort Belvoir, VA 22060

Gentlemen:

The U. S. Department of Energy (DOE) has requested that Exxon Research and Engineering Company (ER&E) forward an eighty-three (83) barrel sample of liquid product, having an initial boiling point within the range from about 350°F to about 450°F and a final boiling point within the range from about 750°F to about 850°F produced in the EDS Process, to your company (U. S. Army) for testing as a diesel fuel. We understand that the sample will be tested to determine its suitability as a diesel fuel at no cost to ER&E or DOE.

ER&E is willing to forward this sample to you pursuant to DOE's request if the U. S. Army agrees as follows:

1. To perform, to the extent not otherwise prohibited herein, tests and physical/chemical characterizations required to determine the suitability of the sample as a diesel fuel;

2. To not perform any tests or procedures which would permit the U. S. Army to determine the concentration of any hydroaromatic component in said sample or any fraction or portion thereof or to identify the amount of donatable hydrogen from hydroaromatics contained in said sample or any of its fractions;

3. To not make the sample or any portion thereof available to any third party. (NOTE: This excludes the U. S. Army Fuels and Lubricants Research Laboratory located at Southwest Research Institute which is a dedicated contractor facility considered to be an extension of MERADCOM's in-house capabilities and which will be bound by the provisions of this Agreement);

4. To not publish any results which would permit anyone to identify the amount of donatable hydrogen therein;

5. To return all unused portions of the sample to ER&E, at ER&E's expense, after the required testing has been completed under the provisions of paragraph 1 above, unless specifically requested to do so otherwise by ER&E or DOE;

6. To furnish to ER&E and DOE the results and conclusions of the tests the U. S. Army conducts on the samples; and

7. To provide an opportunity for ER&E to comment on any presentations made to others of such results and conclusions within two (2) years of the date of this letter agreement;

8. The U. S. Army understands that the products from the EDS Process made available to the U. S. Army hereunder are experimental products and the U. S. Army therefore agrees to indemnify and hold ER&E harmless from all claims, causes of action, demands, losses and damages arising (a) from the injury to or death of employees and agents, except such as may result wholly from the negligence or willful misconduct of ER&E or its employees; and (b) from loss of or damage to property of the U. S. Army or the U. S. Army Fuels and Lubricants Research Laboratory, which results from the U. S. Army's testing of the products made available hereunder.

We trust you will find the foregoing terms acceptable and will so indicate by having two copies of this letter agreement executed on behalf of the U. S. Army in the space provided below. After execution, please return the two executed copies to us for execution on behalf of ER&E. We will then forward one fully executed copy of the agreement and the sample to you.

Very truly yours,

EXXON RESEARCH AND ENGINEERING COMPANY

By Original Signed

ACCEPTED AND AGREED TO:

U. S. ARMY MOBILITY EQUIPMENT RESEARCH
AND DEVELOPMENT COMMAND (MERADCOM)

By Original Signed
Maurice E. LePera

Date
10/25/82
WH:ng

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